## NASA TECHNICAL **MEMORANDUM**

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DEVELOPMENT AND EVALUATION OF AN ABLATIVE CLOSEOUT MATERIAL FOR SOLID ROCKET **BOOSTER THERMAL PROTECTION SYSTEM** 

By W. J. Patterson Materials and Processes Laboratory

October 1979

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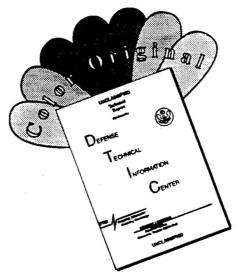
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			evaluated by mechanical property testing as well as thermal testing in a wind				
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#### TECHNICAL MEMORANDUM

## DEVELOPMENT AND EVALUATION OF AN ABLATIVE CLOSEOUT MATERIAL FOR SOLID ROCKET BOOSTER THERMAL PROTECTION SYSTEM

### I. INTRODUCTION

Early in the development of the Thermal Protection System (TPS) for the Solid Rocket Booster (SRB) it was recognized that an easily applied, trowellable closeout/repair material would be required to augment the primary TPS materials, MSA-1¹ and sheet cork. This material must serve to fill in gaps or discontinuities in the primary TPS and must also be applicable in place of the primary TPS in small, poorly accessible areas.

Finally, the closeout material must be useful to repair damaged areas in the primary TPS. The initially defined closeout material goals for SRB flight hardware are summarized as follows:

- 1) Thermal/ablative performance equivalent to surrounding primary TPS
  - 2) Substrate adhesion minimum 100 psi flatwise tensile strength
- 3) Strain compatibility no material failure at a minimum substrate strain of 1.4 percent.
- 4) Processing characteristics readily hand trowellable, thixotropic, long working life, cure in 24 h at 75°F in thick sections
  - 5) Non-flammable per NHB 8060.1A
  - 6) Compatible with primary TPS, topcoats, and substrate paints
  - 7) Removable with high pressure water without substrate damage.

An in-house program was then initiated to develop and qualify such a material for flight hardware application.

<sup>1.</sup> MSA-1 is the Marshall Sprayable Ablator which is applied to nose cap, frustum, and forward skirt elements while sheet cork is applied to the aft skirt.

### II. APPROACH

The basic approach to development of a closeout material was to utilize the excellent adhesive strength and room temperature cure characteristics of epoxy resins, and modify this system with polysulfide to improve flexibility and strain compatibility.

### A. Formulation Development

The bisphenol A - glycidyl ether epoxy chosen for this development was Epon 828, a commercial liquid epoxy prepolymer having a number average molecular weight  $(\overline{\rm M}_{\rm n})$  of 369 and an epoxide equivalent weight of approximately 185, assuming difunctionality of the terminal epoxide groups:

Epon 828 Epoxy Prepolymer

Similarly, the polysulfide prepolymer has a  $\overline{M}_n$  of 1000 and is difunctional with respect to mercaptan terminating groups:

$$\mathsf{HS} \xrightarrow{\hspace{1cm}} \mathsf{CH}_2 \mathsf{CH}_2 \mathsf{OCH}_2 \mathsf{OCH}_2 \mathsf{CH}_2 \mathsf{-S-S} \xrightarrow{\hspace{1cm}} \mathsf{CH}_2 \mathsf{CH}_2 \mathsf{OCH}_2 \mathsf{OCH}_2 \mathsf{CH}_2 \mathsf{-SH}$$

### LP-3 Polysulfide Prepolymer

The mercaptan group is nucleophilic in nature and will slowly attack the primary carbon of the oxirane ring:

However, the reaction rate at room temperature is low and only proceeds at a practical rate by the addition of a base to accept the proton and generate the more strongly nucleophilic mercaptide anion. In this study, several organic bases or amines were investigated to serve as crosslinking agents for the epoxy prepolymer and to promote the epoxide-mercaptan chain extension reaction.

The amines which were screened as potential catalysts or cross-linking agents are as follows:

1) 
$$(CH_3)_2N CH_2 \longrightarrow CH_2 N (CH_3)_2$$
  
 $CH_2 N (CH_3)_2$ 

Tris (dimethylaminomethyl) phenol (DMP-30)

2) 
$$H_2^{N} \bigodot^{NH_2}$$
  $H_2^{N} - \bigcirc - CH_2 - \bigcirc - NH_2$  Methylenedianiline (MPDA) (MDA)

(This was used as 70/30 mixture MPDA/MDA, available commercially as Shell Z)

3) 
$$H_2N CH_2CH_2 N CH_2CH_2 N CH_2CH_2NH_2$$

Triethylenetetramine (TETA)

4) 
$$H_2N - \langle S \rangle - CH_2 - \langle S \rangle - NH_2$$

Bis (4 - aminocyclohexyl) methane (PACM-20).

The tertiary amine (1) acts as an anionic initiator for ring-opening polymerization via the oxirane rings of the epoxy resin, while amines (2) through (4) possess active hydrogen functionality, and serve as authentic crosslinking agents for the epoxy prepolymer.

Substrate adhesion was considered a critical parameter for the closeout material, so the lap shear tensile adhesive strength of the candidate formulations was utilized as an initial screening test. Weight ratios of epoxy/polysulfide of 1/1 were reported [1,2] to provide a

desirable balance of tensile strength and elongation, and this ratio was used as the basis for the formulation studies. The epoxy/polysulfide blend was mixed separately with amines (1) through (4) at concentrations based on the epoxy resin weight. Lap shear tensile adhesive specimens were prepared and cured for 5 days at 70° ± 5°F. Adhesive bond strengths were then determined at 70°F and 300°F² with the results summarized in Table 1.

TABLE 1. LAP SHEAR TENSILE STRENGTH OF CANDIDATE CLOSEOUT RESIN FORMULATIONS

		Lap Shear Adhesive Strength (psi)	
Resin Composition	Catalyst (%)	75°F	300°F
1/1 Epoxy/Polysulfide	(1), 8	1955	0
1/1 Epoxy/Polysulfide	(2), 20	1722	66
1/1 Epoxy/Polysulfide	(3), 10	1140	18
1/1 Epoxy/Polysulfide	(4), 25	1465	84

The tertiary amine designated as (1) resulted in essentially no strength at 300°F and was excluded from further consideration. A second screening criterion employed at this point was the 2 hr pot life requirement. The aliphatic polyamine (3) produced considerable exotherm in the crosslinking reaction due to its relative strength as an organic base and resultant nucleophilic reactivity toward the oxirane ring of the epoxy resin. As a result, the working or application life of the mix was limited to 1/2 hr or less. This deficiency was also observed for catalyst (1). In contrast, the resin catalyzed with the aromatic amine mixture (2) remained workable for over 2 hr. This observation is consistent with the weakly basic nature of aromatic amines. Catalyst (4) was characterized by an application life of 1 to 1.5 hr. Although this is an aliphatic amine, the steric hindrance of the bulky cyclohexyl group reduces the effective nucleophilicity.

The low reactivity of the aromatic amine catalyst, while providing for excellent application life of the closeout material, also results in a need for extended cure time at room temperatures. This is impractical from a production hardware standpoint, and the formulation phase of the program was expanded to assess the effect of accelerators to shorten the overall aromatic amine cure time without seriously compromising the desirable application life. Stannous octoate,  $Sn(O_2C-CH_2)_6$   $CH_3)_2$ , was

<sup>2. 300°</sup>F is the maximum allowable temperature for the SRB aluminum substrate.

chosen for these tests based on its selective accelerating effect on the rates of certain nucleophilic displacements such as the amine/oxirane reaction. The relative rate enhancement due to the accelerator was monitored by viscosity change as a function of time. The test formulations are tabulated in Table 2 and the viscosity data are summarized graphically in Figure 1.

TABLE 2. TEST FORMULATIONS FOR CURE ACCELERATION STUDIES

Formulation	Epon 828 <sup>a</sup>	LP-3	Shell Z	Tin Octoate
1	50	50	10	0
2	50	50	20	0
3	50	50	20	1
4	50	50	20	2
5	50	50	10	3

a. Numbers in each column are parts by weight

As illustrated in Figure 1, the viscosities of formulations 4 and 5 are reasonably independent of time for the first 2 hr (providing for extended working life) but increase sharply from that point on to result in a moderately efficient cure rate. After 48 hr, formulations 4 and 5 had cured to a tack-free, tough, crosslinked solid.

The next stage in formulation development was selection of a low density filler to reduce the overall closeout material density to 30 to 35 lb/ft<sup>3</sup> and provide acceptable thermal/ablative protection. The candidate filler materials were phenolic microballoons, glass microballoons, and ground cork. These fillers were formulated into test closeout mixes to determine maximum filler loading consistent with acceptable trowelling characteristics. This was found to be 40 percent, 30 percent, and 15 percent by weight (based on combined resin weight, 828 + LP-3) for the phenolic, glass, and cork fillers, respectively. The phenolic-filled formulation provided lower density, ease of hand application (trowelling). non-slumping (thixotropic) on vertical surfaces, and cure in thick sections (up to 2 in.) without cracking. Thus, the most promising formulation at this stage of development was the phenolic microballoon filled, aromatic amine/tin octoate cured, epoxy-polysulfide material represented by the compositions shown in Table 3, and the general formulation was given a tentative designation of Marshall Trowellable Ablator, MTA-2.

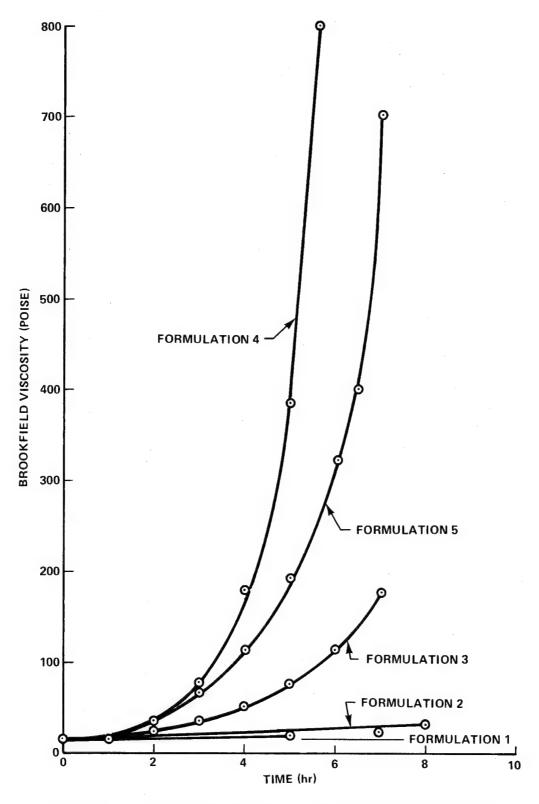


Figure 1. Effect of tin octoate accelerator on cure rates of closeout formulations.

TABLE 3. CANDIDATE MTA-2 CLOSEOUT MATERIAL COMPOSITIONS

	Component Parts by Weight		
Component	Formulation A	Formulation B	
Epon 828	50	50	
LP-3	50	50	
Shell Z	20	10	
Tin Octoate	2	3	
Phenolic Microballoons	40	40	

### B. Closeout Formulation Test and Evaluation

The primary screening parameters for the candidate closeout material were thermal/vacuum exposure and flatwise tensile testing which focused primarily on the MTA formulations. The thermal/vacuum test specimen consisted of 1/2-in. cork bonded to aluminum substrate. A 2-in. wide groove was cut to the substrate to receive the closeout material. The thermal exposure consisted of a constant radiant heating environment of 15 Btu/ft<sup>2</sup>-sec for 130 sec, with a simultaneous pressure decay to simulate the SRB ascent environment. The specimens were instrumented to monitor substrate temperature. Figure 2 illustrates the thermal/vacuum closeout specimen.

The primary determinants in this type of test were char depth (recession) and char integrity. The goal was to obtain a recession rate no greater than the surrounding primary TPS and to produce a tough, stable char to enhance insulating quality. The MTA formulations identified in Table 3 performed quite well in this test as indicated in Figures 3 and 4. These figures verify that the closeout material char depth approximates that of the surrounding cork TPS. By comparison, the recession rates of a commercial cork-filled ablator (Fig. 5) and the aliphatic amine cured ablator (Fig. 6) were substantially higher as summarized in Table 4.

The flatwise tensile strength measurements were performed at 75°F and 300°F utilizing a specimen configuration illustrated in Figure 7 and pulled in tension to failure on an Instron tester. The failure mode was consistently cohesive (bulk failure in the material) at both temperatures for the candidate formulations, as summarized in Table 5. Again, the beneficial effects of the aromatic amine crosslinking agent are manifested in the 300°F flatwise tensile strength.

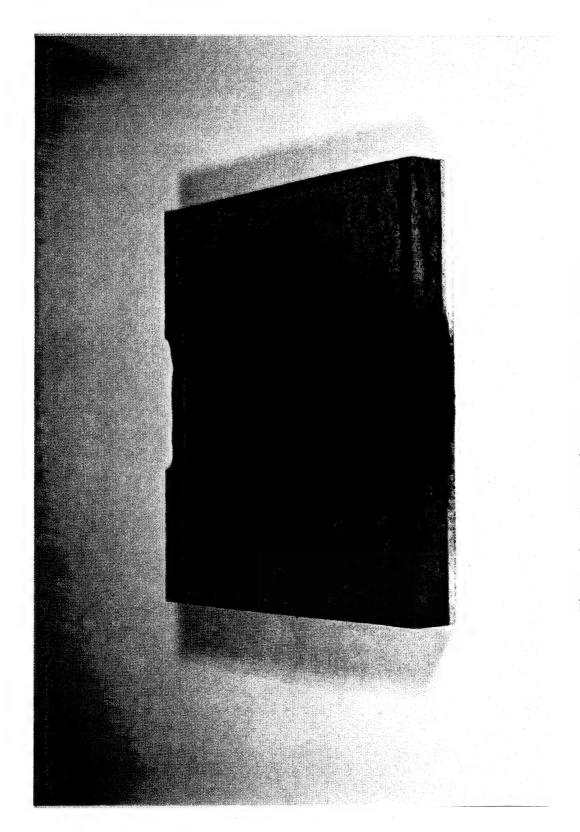


Figure 2. Thermal/vacuum closeout specimen.

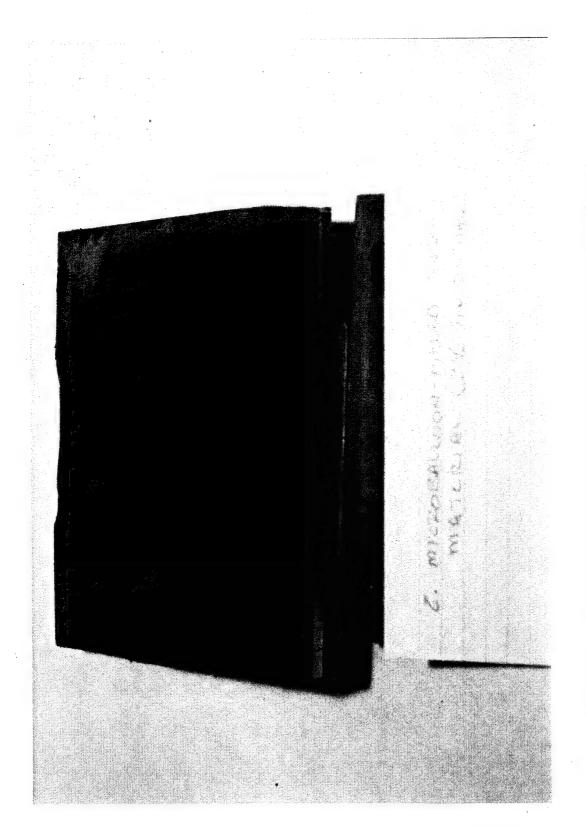


Figure 3. Post-test thermal/vacuum specimen, MTA-2 (Formulation A).

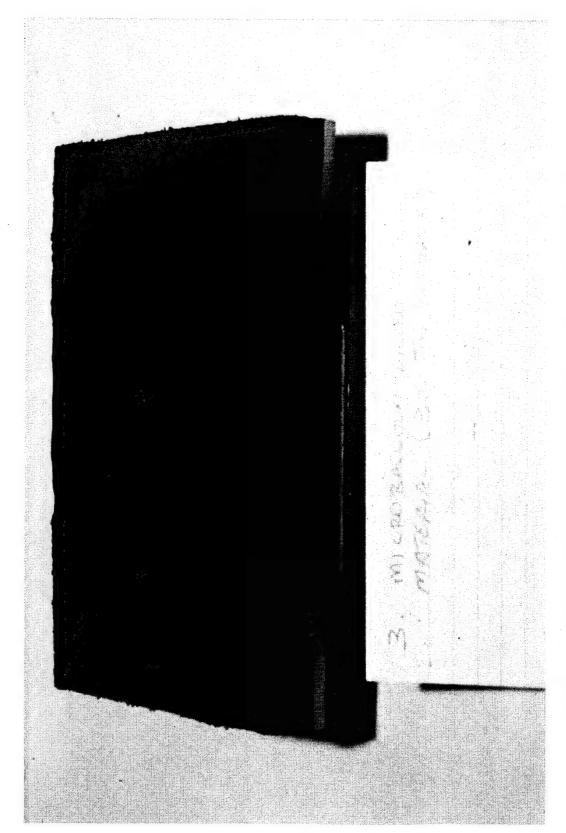


Figure 4. Post-test thermal/vacuum specimen, MTA-2 (Formulation B).

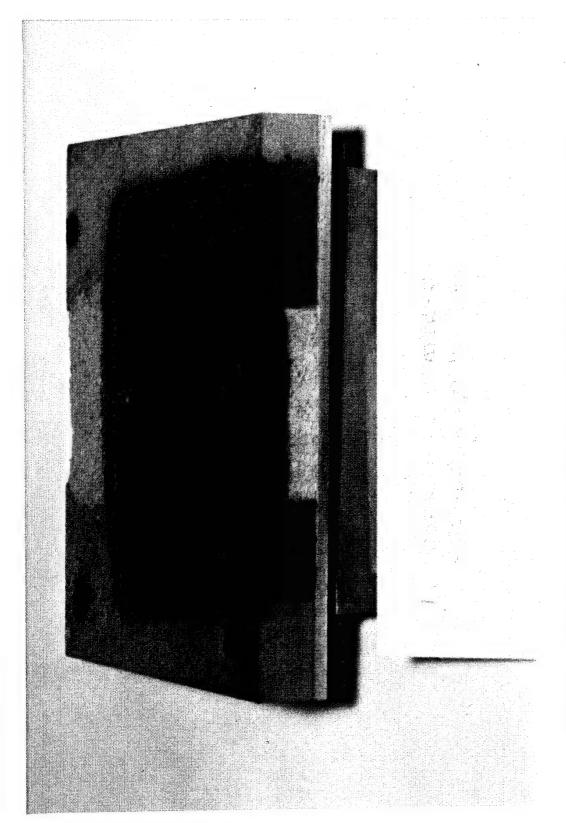


Figure 5. Post-test thermal/vacuum specimen, cork-filled epoxy formulation.

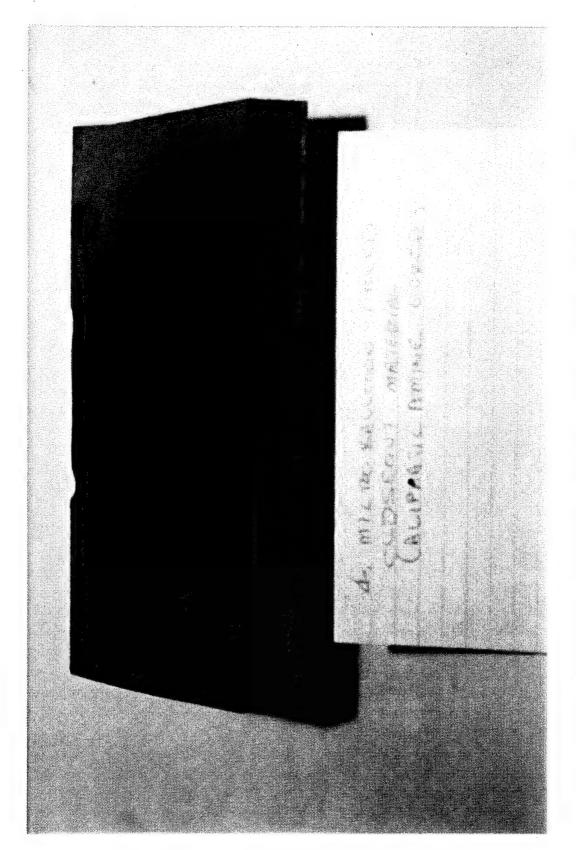


Figure 6. Post-test thermal/vacuum specimen, aliphatic amine cured MTA formulation.

TABLE 4. THERMAL/VACUUM TEST RESULTS FOR CANDIDATE CLOSEOUT FORMULATIONS

Formulation	ΔT <sup>a</sup> (°F)	Char Depth <sup>b</sup> (percent)	Char Condition
MTA (A)	113	40	Tough, stable char
MTA (B)	118	50	Tough, stable char
Aliphatic Amine Cured MTA	119	60	Weak, loose char
Cork Filled Epoxy	212	90	Very fragile char, substrate visible

- a.  $\Delta T$  = Substrate temperature rise above 75°F.
- b. Percentage of total specimen thickness converted to char, based on original thickness of 1/4 in.

TABLE 5. FLATWISE TENSILE STRENGTHS OF CANDIDATE CLOSEOUT FORMULATIONS

·	Flatwise Tensile	Strength (psi)
Formulation	75°F	300°F
MTA-2 Formulation A	625	72
MTA-2 Formulation B	510	49
Aliphatic Amine Cured MTA	386	17
Cork-Filled Epoxy	404	24

The cure time characteristics of the candidate MTA-2 closeout formulations were monitored by hardness techniques. Shore hardness measurements were made as a function of time at a given temperature to establish minimum cure time, defined as the time required to reach an essentially constant hardness. The MTA-2 formulations A and B reached a cured condition after a minimum of 24 hr at 100° to 105°F, as opposed to 48 and 72 hr for formulations A and B, respectively, when cured at

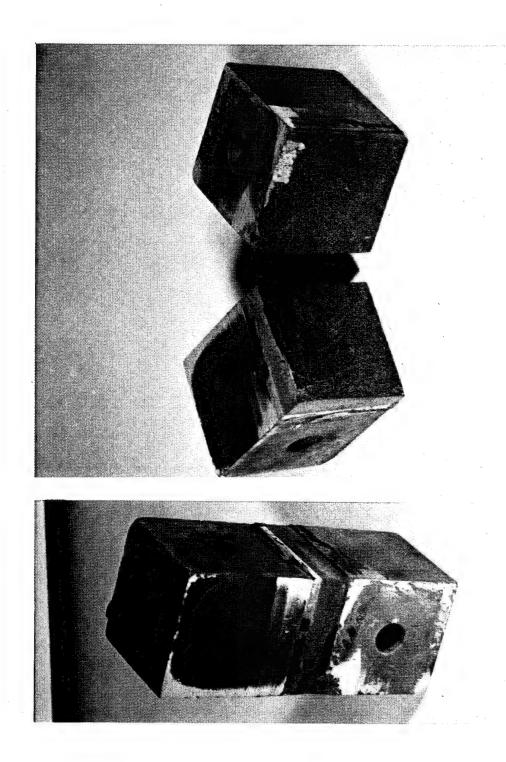


Figure 7. Flatwise tensile strength specimen of closeout material.

temperatures as low as 65°F. At cure temperatures as low as 40°F, the specimens required a minimum of 144 hr to reach an equivalent hardness. In each case, the adequacy of cure was verified by flatwise tensile strength measurements (minimum 100 psi requirement). The cure time/temperature data for the MTA-2 formulation A are summarized in Table 6.

TABLE 6. CURE TIME VERSUS TEMPERATURE FOR MTA-2 CLOSEOUT FORMULATION A

Cure Time (hr)	Temperature (°F)	Flatwise Tensile Strength (75°F)
24	40	Material soft, uncured
48	40	Material soft, uncured
72	40	Material firm, partially cured
144	40	242 psi
24	70	82 psi
48	70	466 psi
24	85	189 psi
24	100	480 psi

### III. EVALUATION TESTING

### A. Thermal Property Assessment

Based on the development test results discussed in the preceding section, the decision was made to go into detailed thermal testing with the MTA-2 formulation (A), now designated simply as MTA-2, although formulation (B) was also carried through portions of this test phase. The principal thermal testing was performed in the MSFC Modified Hot Gas Facility (MHGF). The MTA-1 assessment included testing as a closeout/repair material for both MSA-1 primary ablator (for forward SRB elements with lower heating rate) and for sheet cork on the SRB aft skirt where a higher thermal environment is experienced. The MHGF wind tunnel test panels for cork closeout evaluation were approximately 21 by 27 in. Sheet cork, 1/2-in. thick, was bonded to the

aluminum substrate and 2-in. grooves were milled down to the substrate to produce a closeout test pattern in the cork. Bolts were located along the grooves to simulate fasteners on the SRB flight hardware. These were encapsulated with PR-1422 polysulfide sealant and fitted with a sealant cap. This again reflects the flight hardware fastener configura-The closeout material was applied to the test panel and allowed to This panel buildup sequence is illustrated in the series of Figures 8, 9, and 10. The panel was instrumented with thermocouples on the dummy fasteners and on the substrate. This test panel was configured to allow simultaneous test of two closeout materials, each occupying one half of the panel. Figure 11 illustrates the final pre-test condition with Figure 12 showing the effects of a 60-sec run in position 3 of the MHGF with a heating rate distribution of 20 to 44 Btu/ft<sup>2</sup>-sec from leading to trailing edge of the panel. As indicated from Figure 12, the MTA (upper half of closeout pattern) performed quite well with adequate char remaining to provide thermal protection to the fasteners and over all closeout No substrate or fastener temperatures above 90°F were observed. The lower half of the closeout pattern was MTA cured with the aliphatic amine, PACM-20. The higher recession rate is obvious from exposure of the simulated fasteners, and is believed to result from the relatively poorer char stability typically conferred by aliphatic amine cured systems. A further example of the utility of MTA-2 as a cork closeout is illustrated by Figure 13. This specimen is a segment of a MHGF panel run under conditions described earlier. The cross section vividly shows the close correspondence of cork and MTA-2 recession rates, which constituted a critical goal of this development program.

In this phase of the development testing, several MHGF runs were made to define appropriate closeout configuration and thickness. For example, in Figure 14 the MTA-2 closeout material was applied flush with the cork in the circular repair simulation patterns, and just thick enough to cover the fastener caps (approximately 1/8 in. coverage) with essentially no ramping of the MTA-2 out onto the adjacent cork on the leading As a consequence, the repair area survived quite well (no interference heating effects), but the fastener area acted as sufficient proturberance to cause high localized heating and expose the fasteners. Two alternate configurations were then evaluated. In the first of these (Fig. 15), the MTA-2 was built up to a closeout thickness that allowed a minimum of 1/2 in. above the top of the fastener and ramped down 4 in. from the fastener center line onto the adjacent cork surface. second configuration involved cork strips on either side of the fastener row forming a trough which was filled with MTA-2 level with the cork. Also the leading edge was ramped down at 30 degrees to the adjacent cork (Fig. 16). The post-test photograph (Fig. 17) shows adequate retention of closeout material to protect the fasteners (fastener on near

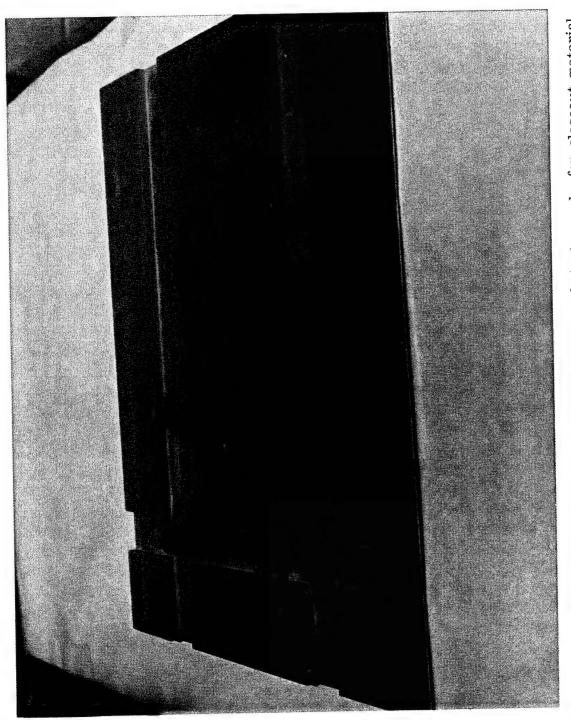
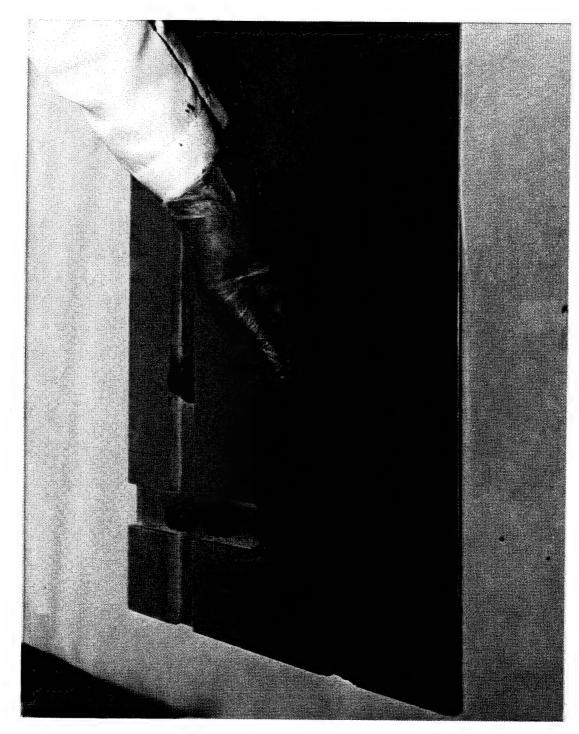


Figure 8. MTA-2 closeout panel buildup sequence: substrate ready for closeout material.



MTA-2 closeout panel buildup sequence: initial application. Figure 9.



Figure 10. MTA-2 closeout panel buildup sequence: final surface finishing of closeout.



Figure 11. Pre-test MHGF panel of MTA-2 closeout material.

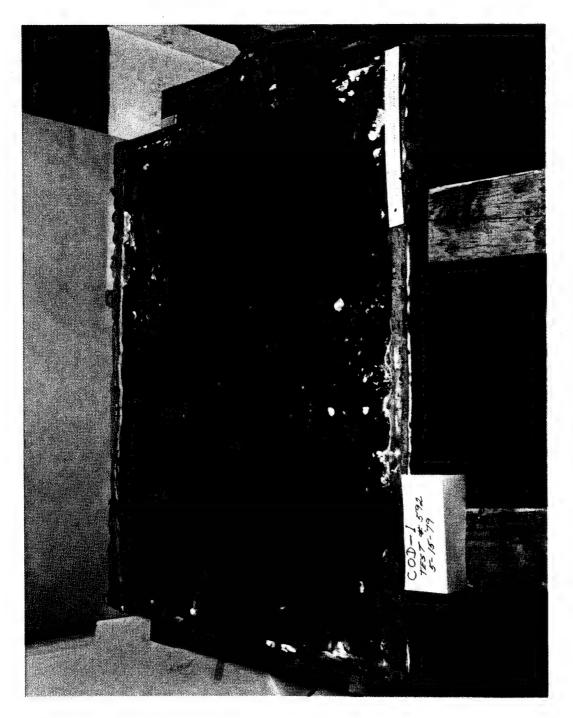


Figure 12. Post-test MHGF panel of MTA-2 closeout material.

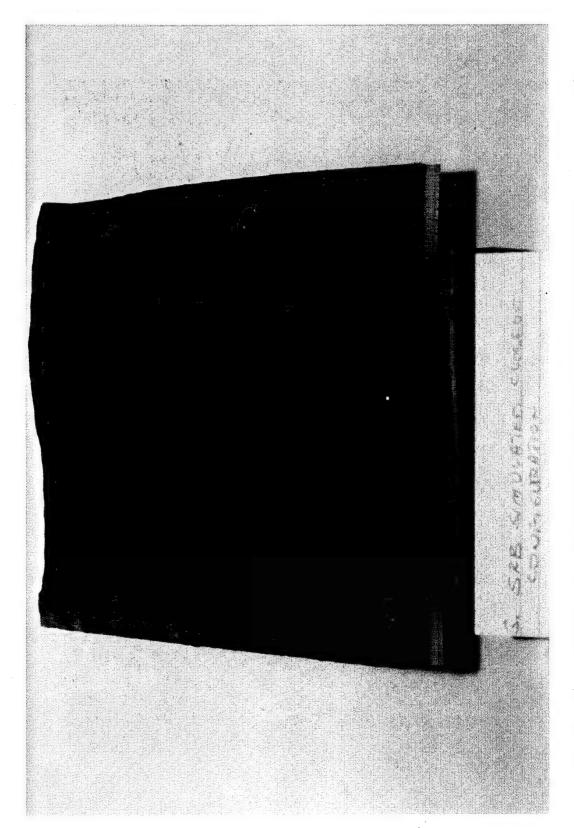


Figure 13. Post-test MHGF closeout panel cross-section.

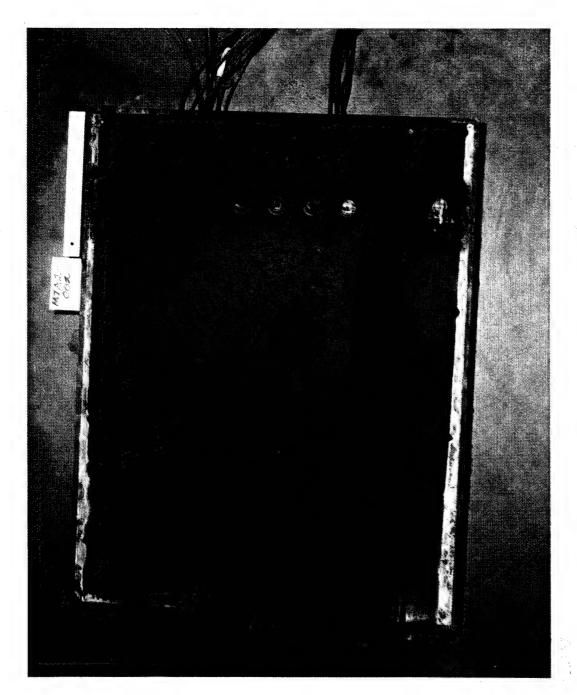


Figure 14. Post-test MHGF panel: closeout thickness assessment.

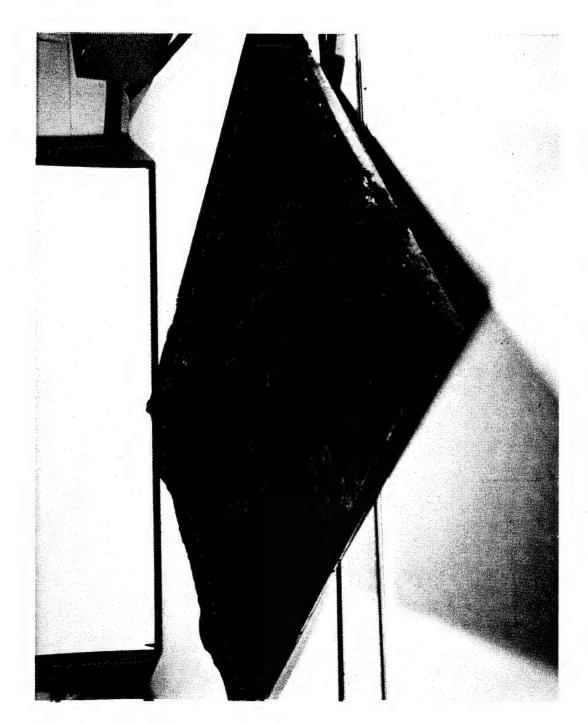


Figure 15. Post-test MHGF panel: closeout thickness assessment.

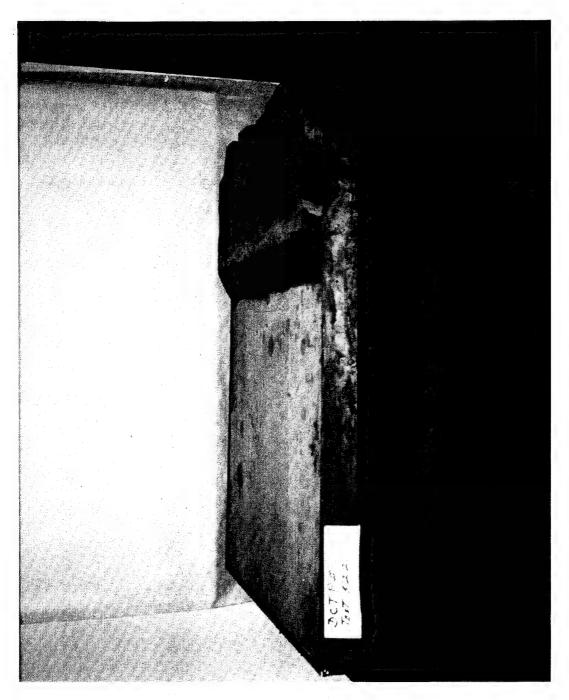


Figure 16. Pre-test MHGF panel: closeout configuration assessment.



Figure 17. Post-test MHGF panel: closeout configuration assessment.

edge of panel was exposed deliberately from pre-test panel fitting operation). Both closeout configurations performed successfully with no significant substrate heating.

To evaluate the use of MTA-2 with the MSA-1 primary ablator, MHGF panels sprayed with MSA-1 and coated with white topcoat were modified with MTA-2 closeout/repair areas as shown in Figure 18. These panels were run in position 2 of the MHGF for 60 sec (heating rate range for position 2 is 8.2 to 20 Btu/ft<sup>2</sup>-sec). The five repair areas are visible in Figure 19 as the dark circles. The MTA-2 recession was less than that of the surrounding MSA-1, although it is not obvious from the angle of the photographic view. The corresponding Figure 20, in which the char layer on this panel was removed down to virgin ablator, verifies the acceptable performance of the MTA-2.

The closeout material is expected to have a number of specialized applications on protuberances where high aeroshear-induced heating rates are found. The MTA-2 was tested in two such protuberance configurations which further reinforced the confidence in its thermal/ablative performance. Figure 21 shows an SRB kick ring model in which MTA-2 was used to protect fasteners on the top surface of the model. After a 17.1-sec run at 30 to 40 Btu/ft<sup>2</sup>-sec, the MTA-2 was still intact and provided adequate thermal protection for the fastener (Fig. 22). In the second protuberance application, MTA-2 was utilized to ramp up from adjacent cork TPS to the top of an instrumentation island (the MTA-2 is shown painted white with the surrounding cork in Fig. 23). This specimen was run at the Arnold Engineering Development Center wind tunnel, Tullahoma, Tennessee, and exposed to 8.6 to 13.5 Btu/ft<sup>2</sup>-sec for 107 sec. The post-test condition shown in Figure 24 indicates adequate char stability and ablative performance.

An extremely severe SRB thermal environment is encountered for very short times (less than 10 sec) as the separated SRB falls back through the Shuttle main engine exhaust plume. Special tests were conducted at Aerotherm, Inc., Mountain View, California, in which the MTA-2 was exposed to severe heating in the range of 72 to 138 Btu/ft<sup>2</sup>-sec. The results, summarized in Table 7, indicate that even at 136 Btu/ft<sup>2</sup>-sec, the sample survives approximately 9 sec before the 300°F substrate limit is reached.

<sup>3.</sup> Variations of these configurations were ultimately implemented on the SRB aft skirt in areas of high heating rates.

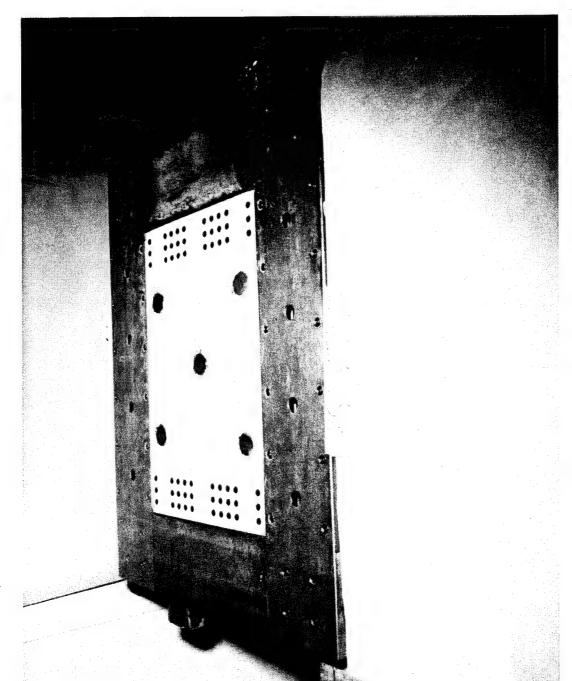


Figure 18. Pre-test MHGF panel: MTA-2 closeout on MSA-1 spray ablator.

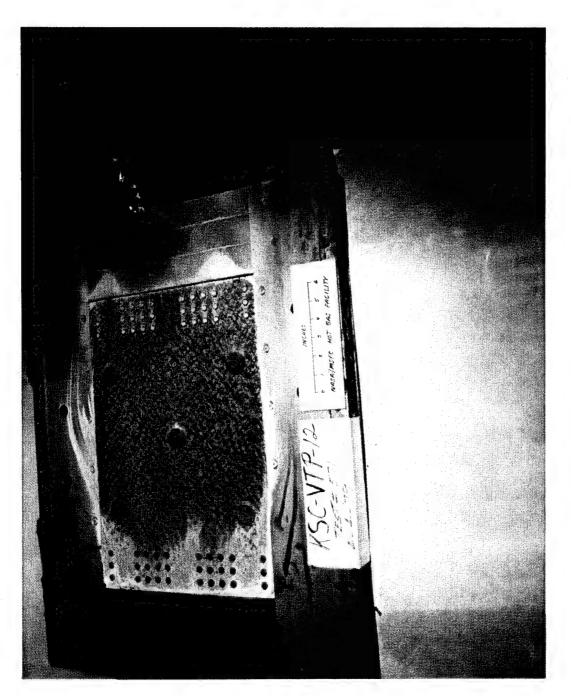


Figure 19. Post-test MHGF panel: MTA-2 closeout on MSA-1 spray ablator.

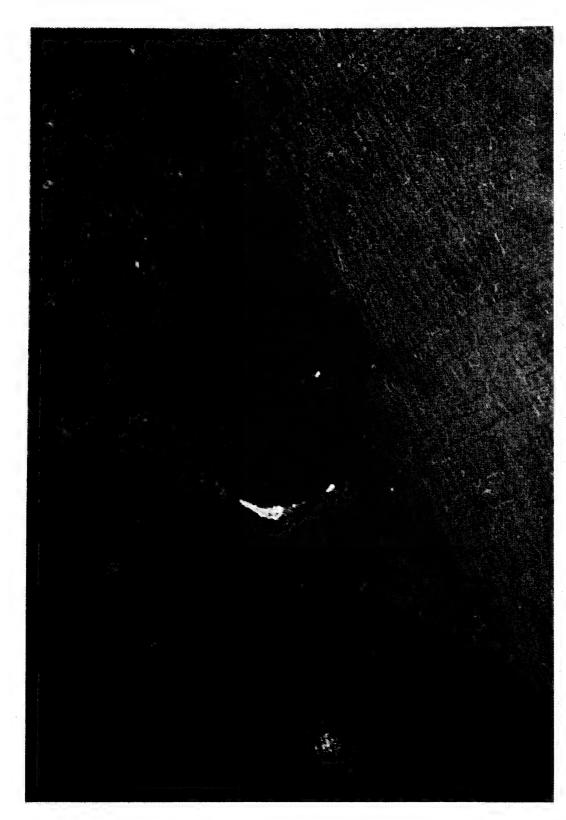


Figure 20. Post-test MHGF panel: MTA-2 closeout on MSA-1 spray ablator.



Figure 21. Pre-test MHGF kick attach model with MTA-2 fastener insulation.



Figure 22. Post-test MHGF attach ring model with MTA-2 fastener insulation.

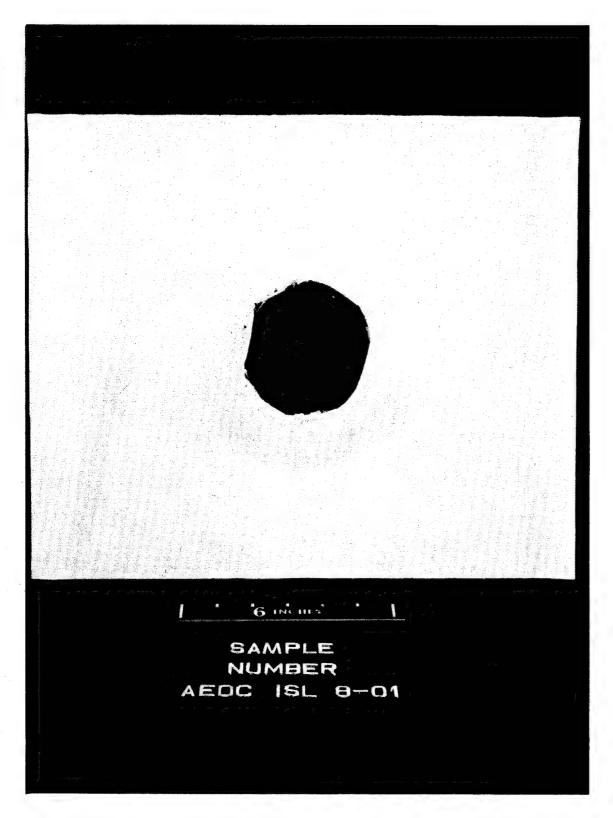


Figure 23. Pre-test AEDC instrument island model with  $$\operatorname{MTA-2}$$  closeout.

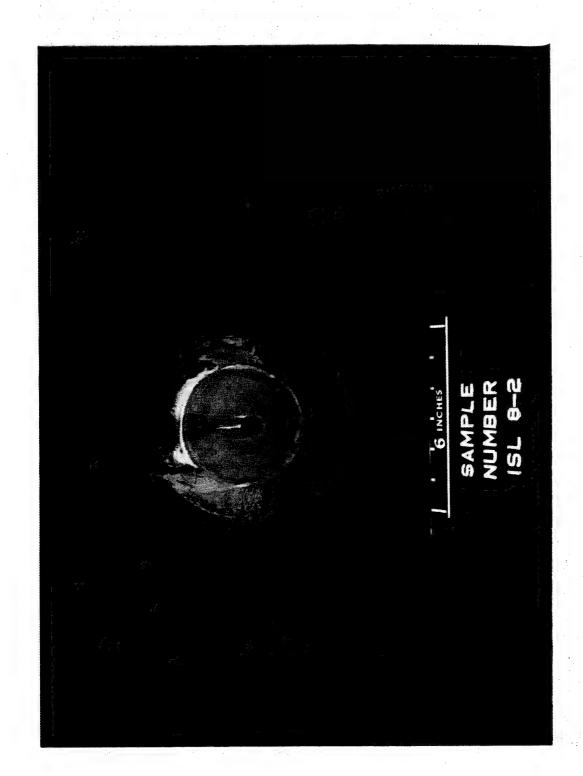


Figure 24. Post-test AEDC instrument island model with MTA-2 closeout.

TABLE 7. MTA-2 THERMAL TEST IN SIMULATED SSME ENGINE PLUME IMPINGEMENT ENVIRONMENT

Test	Heating Rate (Btu/ft <sup>2</sup> -sec)	Exposure Time (sec)	Back Face Temperature (°F)
1	72	16	285
2	77	21.1	217
3	136	9.4	307
4	138	5.9	140

# B. Physical/Mechanical Property Assessment

The following properties of MTA-2 were determined as part of the overall qualification assessment.

1. Flatwise tensile strength. Test specimens 2 by 2 in. were cut from a previously cured 1/2-in. thick trowelled test panel. Flatwise tensile specimens were prepared and pulled in the Instron tester at a cross head speed of 0.050 in./min, at 75°F and 300°F. Peak load at break was used to determine flatwise tensile strength. The final qualification values for MTA-2 are given in Table 8, and represent an average of 4 individual data points in each case.

TABLE 8. FLATWISE TENSILE STRENGTH ASSESSMENT DATA FOR MTA-2

	Cure Time	Cure Temperature	Flatwise Tensile Strength (psi)	
Formulation	(hr)	(°F)	75°F	300°F
$MTA-2 (A)^{a}$	48	70	425	52
MTA-2 (A)	24	100	565	39
MTA-2 (B) <sup>b</sup>	72	70	495	49
MTA-2 (B)	24	100	601	55

a. 50 parts 828/50 parts LP-3/20 parts Shell Z/2 parts tin octoate.

b. 50 parts 828/50 parts LP-3/10 parts Shell Z/3 parts tin octoate.

- 2. Density. Density was determined on 2 by 2 by 1/2 in. thick trowelled specimens by calculation from weight and volume measurements. The average density based on 4 individual determinations was  $30.5 \text{ lb/ft}^3$ .
- 3. Flammability. A set of three flammability specimens was prepared by trowelling 1/2-in. thick MTA-2 onto 12 by 2 1/2 in. aluminum substrates. The specimens were tested in accordance with NHB 8060.1A, Test 1, for upward flame propagation in air. The samples self-extinguished within 6.5 in. (specification requirement is 6 in.). The MTA-2 was judged acceptable by material useage agreement.
- 4. Moisture Absorption. Specimens of MTA-2, 2 by 2 by 1/4 in. thick were weighed on an analytical balance, totally immersed in distilled water for 24 hr, dried of surface moisture, and reweighed. The average (4 specimens) weight gain was 2.27 percent. These specimens were then bonded into flatwise tensile specimens and tested at 75°F. An average strength of 512 psi was measured, which represents a 9.4 percent decrease attributable to water exposure based on the intial strength data cited in Table 8.
- 5. Strain Compatibility. The strain compatibility of the MTA-2 closeout material was determined in accordance with the protocol and procedure in Reference 3. The simulated MSA-1/MTA-2 closeout was made in the gage section of the tensile specimen shown in Figure 25. The specimen was subjected to a substrate strain in excess of 1.4 percent before failure occurred. This failure was in the form of a transverse crack progressing uniformly through both MSA-1 and MTA-2 (see arrow in Fig. 25).
- 6. Removal Assessment. Test panels containing 2-in. strips of MTA-2 closeouts were run in the MHGF to generate a charred surface simulating the TPS condition on recovered SRB hardware. The MTA-2 closeout strips were removable with high pressure water (hydrolaser, 8,000 to 10,000 psi) with no significant damage to the underlying Bostik paint.

### IV. CONCLUSIONS

Based on the results of the evaluation testing, the MTA-2 closeout material was judged suitable for use on appropriate areas of the SRB, excluding the high interference heating areas which require a glassphenolic TPS. The actual qualification or verification of the MTA-2 for flight hardware has been performed separately as part of the overall SRB/TPS verification program, and those results will be reported separately. Subsequently, MTA-2 has been called out on appropriate SRB TPS drawings for use on flight hardware. United Space Boosters, Inc. (USBI), the SRB TPS contractor, has utilized input from this development program to prepare the following documentation:

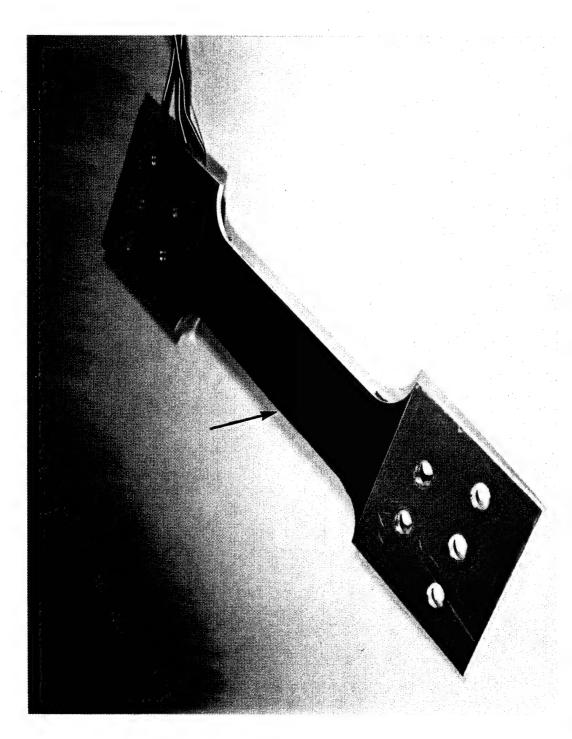


Figure 25. Strain compatibility specimen simulating MSA-1 ablator with MTA-2 closeout.

- 1) USB-MS-2003, "MTA-2 Insulation" (Material Specification).
- 2) USB-PS-1003, "Insulation Application, MTA-2" (Process Specification).

This has been further elaborated by USBI to working-level documents for application to SRB flight hardware:

- 1) STP 433, "MTA-2 Preparation and Mixing," Rev. B, June 1, 1979.
- 2) STP 434, "Application and Finishing of MTA-2 Insulation, Rev. A, October 18, 1978.

At the writing of this report, MTA-2 closeout material had been utilized in the thermal protection system for the first two flight sets of SRB hardware.

Early attempts by USBI to apply MTA-2 to flight hardware led to some discoloration of the white topcoat that is subsequently applied over the primary and closeout TPS. This has been attributed to the excess of Shell Z curing agent used to obtain an efficient curing reaction at room temperatures. The preliminary assessment of the alternate MTA-2 formulation designated as (B) in Table 8 indicates that this problem can be minimized by reduction of Shell Z content concurrent with increasing the concentration of the accelerator, tin octoate. This formulation modification is being pursued as a product improvement activity, together with a detailed assessment of a faster-curing cork-filled epoxy commercial formulation. Results of these studies will be communicated in a subsequent report.

## **REFERENCES**

- H. Lee and K. Neville: Handbook of Epoxy Resins. McGraw-Hill, New York, 1967.
- 2. K. R. Cramker and A. J. Breslau: Industrial and Engineering Chemistry. 47, 98 (1956).
- 3. W. J. Patterson: Strain Compatibility Assessment for SRB Sprayable Ablator MSA-1." NASA TM-78228, April, 1979.

## **APPROVAL**

# DEVELOPMENT AND EVALUATION OF AN ABLATIVE CLOSEOUT MATERIAL FOR SOLID ROCKET BOOSTER THERMAL PROTECTION SYSTEM

By W. J. Patterson

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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